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Credit author statement

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Impact of sodium alginate gelling and ingredient amalgamating order on ingredient interactions and structural stability of ice cream

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Highlights:

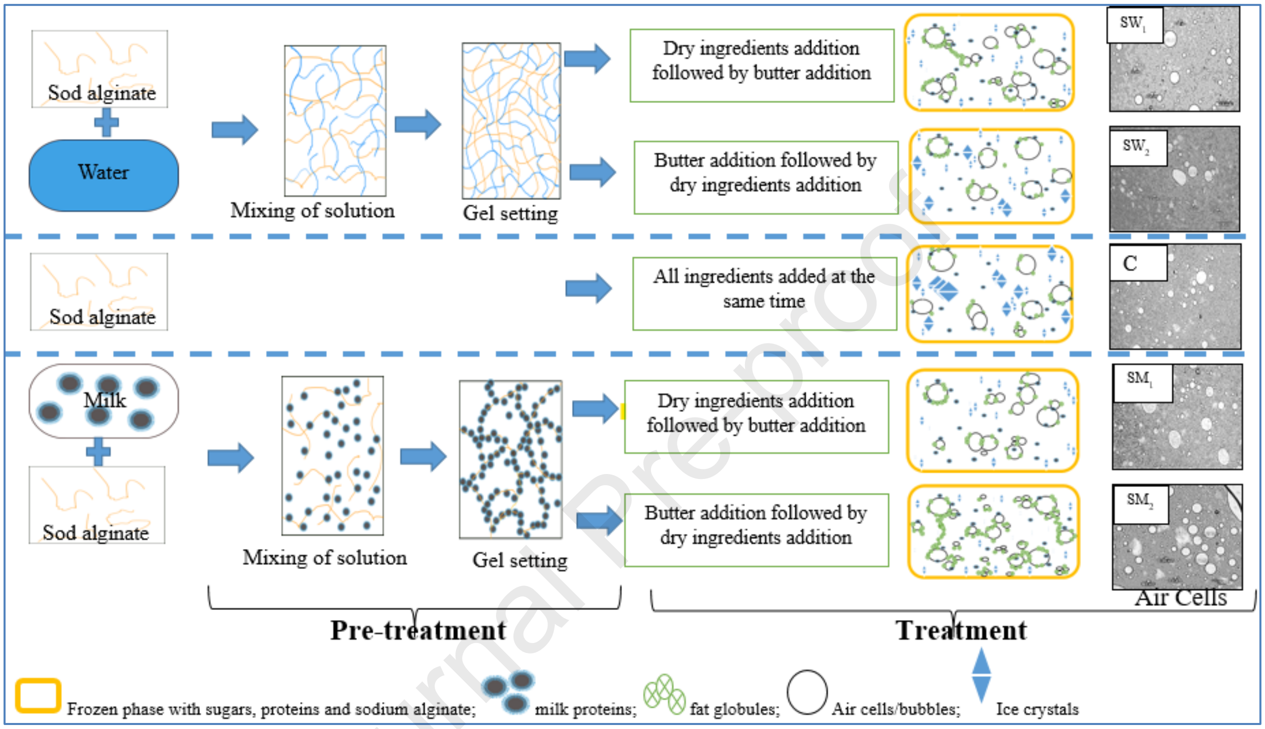
- A standard ice cream recipe was reformulated by amalgamating ingredient addition order.
- Water and milk based sodium alginate gels were developed and used for mix development.
- Physico-chemical properties, sensory, microscopic, and melting characteristics of ice cream samples was studied.
- Reformulation can yield a mix with high stability and overrun, smaller ice crystals and more destabilized fat.

Abstract

The impact of ingredient amalgamating order on the physicochemical properties, sensory, microscopic, and melting characteristics of ice cream was studied. The pre-treatment step involved sodium alginate hydrated in water (SW) and milk (SM). Control (C) sample was developed using standard dairy-based ice cream mix formulation technique whereas SW and SM samples were further mixed with the milk and order of addition of dry ingredients prior to butter addition (SW1, SM1) was compared to the samples in which butter was added before dry ingredients inclusion (SW2, SM2). It was observed that the consistency and structural characteristics of ice cream samples were significantly influenced by pre-treatment and treatment steps. SM1 sample exhibited maximum viscosity; however, SM2 was the most stable formulation with the highest first drip loss time i.e. 25.19 min and melting resistance. Moreover, maximum fat destabilization and overrun in the ice cream manufacturing process was observed in SM2 with the highest overall acceptability score of 8.08 on 9 points hedonic scale. It was found that sodium alginate gel hydrated in milk when added to milk and butter followed by dry ingredients addition forms a superior mix in terms of air cells with strong emulsion boundaries and small ice crystals.

Keywords: ice cream, sodium alginate, ingredient interaction, melting characteristics, microscopic structure

Graphical abstract:



1. Introduction

Ice cream, in the unfrozen state, is a type of emulsion, commonly called as matrix/serum in which continuous serum phase i.e. fat molecules are embedded in a dispersion of water-sugar-ice structure along with air bubbles (Dickson, 2003). Ice cream manufacturing technology has experienced an implausible improvement in terms of structural stability and storage over the years (Goff, 2008). Despite these advancements, the product's stability is challenged by temperature changes during storage and transportation due to enlarging ice particle size, the disproportion of air cells, and water loss from the matrix (Crilly, Russell, Cox, & Cebola, 2008). Recrystallization during freeze-thaw cycles has been associated with the formation of cryo-gels or embroidered networks (Regand & Goff, 2003). And it is a well-known fact that the textural properties of ice cream are the key aspects that determine their market success (Goff & Hartel, 2013). Therefore, ice cream manufacturers are constantly seeking solutions to improve ice cream quality.

It is critical to control ice cream properties while maintaining its structure, texture, and body. A slight change in any step may result in a synergetic effect on the structure of ice cream when used in combination with other components (Syed, Anwar, Shukat & Zahoor, 2018). Therefore, ingredients such as hydrocolloids (alginate, cellulose, etc.) augment the serum-phase micro-viscosity and resist air cells from shrinking during storage. Hydrocolloids have an inequitable impact on ice cream structure, even at low concentrations, due to their different properties (Hagiwara & Hartel, 1995). As hydrocolloids can exhibit their functionality at lower concentrations, the addition of ingredients and mixing techniques can impact the mix consistency. Due to the solubility and gel-setting conditions during freezing and hardness, alginates are preferred for ice creams. Alginate is a non-gelling stabilizer and serves as a thickening agent that provides a broad variety of flow behavioral property i.e. gel firmness, etc., correlated with crystal growth inhibition and changes the morphology (Blond, 1988). In the presence of calcium ions (Draft, 2000), it sets a strong gel structure on cooling which lowers the recrystallization rate than gelling stabilizers (Regand & Goff, 2003). According to Soukoulis, Chandrinos, and Tzia (2008), sodium alginate can control ice recrystallization, regardless of the storage time, and result in improved frozen texture when compared to other gums. The published research in the literature is focused on the effect of an intervention in amalgamating the order of sodium alginate with other ingredients in ice cream. It has been reported that sodium alginate has a synergetic effect in relation to ingredient addition on the consistency of ice cream by binding the free water molecules. Therefore, ice cream manufacturing process requires further improvements especially in terms of ingredients and/or formulation to improve the overall appearance along with physical characteristics of the product.

To the best of the authors knowledge, the order of addition of ingredients has yet not been studied or published in the scientific literature and was identified as an industry requirement for ice cream manufacturers. The present study was carried with the objectives to study (1) the effect of different liquid phases on sodium alginate hydration and impact of ingredient amalgamation order on ice cream quality; (2) hierarchal cluster analysis (Wards method) to study cream stability and (3) correlation analysis between sensory attributes with physical and instrumental properties using Principal Component Analysis (PCA).

2. Materials and methods

2.1. Formulation of the mix

Five types of sample mixes were formulated in 5 kg batches for the final recipe to contain 11% milk fat, 14% sugar, 11% milk solid not fat (MSNF) using the method described by Arbuckle (1997). Milk, butter, and skimmed milk powder (SMP) (Verka Milk Plant, Ludhiana, Punjab, India), sugar, glycerol-mono-stearate (GMS), and sodium alginate (Loba Chemie, Pvt. Ltd., Mumbai, India) were used in ice cream making (Table 1).

2.2. Experimental design

The ice cream development process was split into pre-treatment and treatment as shown in fig. 1 and discssued below:

Pre-treatment

Sodium alginate was blended in three different ways; Control, SW and SM. For 5 kg ice cream mix, sodium alginate powder (17.5 g) was hydrated in 300 ml warm water/milk (Table 1) with continuous stirring using a magnetic stirrer (capacity 2 lt.) on the hot plate (Supertek, India).

Treatments

Pre-treated samples were blended in a different orders to make the final ice cream mix. The control mix was prepared by the standard protocol given by De (2019). In SW and SM samples, further two treatments (Table 1) i.e. reverse order of addition of dry ingredients and butter, were tested for their effect on ice cream properties.

All the treatments were homogenized using a two-stage (single piston) homogenizer at 2000 and 500 psi (Taj make, homogenizer, New Delhi) and pasteurized at 80 °C for 25 sec. Freshly prepared mixes were quenched to 4 °C for about 24 h for aging. To the aged mix, vanilla flavor (bush, artificial vanilla scent agent) was added in the mixing freezer (Sigma Sales Corporation, New Delhi, India), filled in round polystyrene cups (50 ml capacity, Rajshree PolyPack, India) and placed overnight in the deep freezer (Blue Star, India) at -18 to -20 °C for hardening.

2.3. Physico-chemical properties

Total solids, proteins, fats, total minerals, and titrable acidity (as lactic acid) were determined using the AOAC (2000) method in three replicates. Gerber method was used to analyze the amount of fat milk and butter (BIS, 1981). The pH was evaluated using a pH meter (System IQ 125, USA).

2.4. Creaming Stability

Creaming stability was determined using the method described by Cheng, Ma, Li, Yan, & Cui (2015). Aged samples were stored at 4 °C in cork test tubes (H: 150 mm, D: 17 mm) with a capacity of 25 ml for three weeks (for mix stability) to determine creaming index, expressed as serum volume fraction (percent) and calculated using the equation [1]:

$$\text{Serum volume fraction, \%} = \frac{H_e}{H_s} \times 100 \qquad (1)$$

Here, H_e is emulsion separation height (cm) and H_s serum layer height (cm)

2.5. Viscosity

Brookfield Viscometer (Model LVT) was used to carry out measurements at a speed of 60 rpm using spindle no. 3 at 20 ±1°C (Goraya & Bajwa, 2015).

2.6. Specific gravity (SG)

The specific gravity of mix and ice cream was calculated by the method described by Goraya & Bajwa (2018). A cup of known weight (W_1) was filled with water (W_2)/ mix and melted ice cream (W_3) and weighed. The specific gravity of the sample was determined by the following equation [3]**:**

$$SG = \frac{W_3 - W_1}{W_2 - W_1} \tag{3}$$

2.7. Overrun

Overrun was analyzed by comparing the known weight of the mix and ice cream, calculated using the equation [2]:

$$\text{Overrun, \%} = \frac{\text{Mix weight} - \text{Ice cream weight}}{\text{Mix weight}} \times 100 \tag{2}$$

2.8. Hardness

Ice cream cups (-20 °C) were tempered for 4 hours to reach a temperature of -5°C prior to measurement using a texture analyzer (TA-XT plus, Stable Microsystems Ltd., UK). The sample was held horizontally under the Warner / Blazer (HDP / BS) knife-edge blade (soaked in icy water before and after each measurement) using a 5 kg load cell. Force compression setting at a distance of 35 mm, auto-trigger types 20g, and data acquisition rate 250 PPS were set up with a pre-test, test, and post-test speed as 2, 3, and 10 mm/s, respectively. Hardness was analyzed from the compression force (g) peak detected when the blade proceeds to cut the ice cream sample.

2.9. Melting characteristics

First drip time (FDT) min and melting rate (w/w%) were determined to estimate the melting characteristics of samples. Ice cream samples (50g and -18°C) were placed on a wire sieve (6 holes/cm) placed on the top of a graduated cylinder funnel at 20 ± 2°C and measured at an interval of 10 min for 100 min duration (Goraya & Bajwa, 2018).

2.10. Fat Destabilization

Fat destabilization was measured with some modifications in the spectroscopic techniques outlined by Goff & Jordan (1989). Turbidity was taken as an indicator of the amount of fat destabilization at 540 nm and Milli Q water as blank was used to evaluate as equation [4]:

$$\text{Fat destabilization }\% = \frac{\text{A}_{540}\text{ of mix} - \text{A}_{540}\text{ of ice cream}}{\text{A}_{540}\text{ of mix}} \times 100 \quad (4)$$

2.11. Color analysis

The color profile of the samples was measured using Mini Scan Xe Plus (Hunter Color Lab) in triplicate and expressed as ‘L*’, 'a*', and 'b*' representing the color intensity of the product (Singla, Kumar, Kaur & Goraya, 2020).

2.12. Sensory attributes

The ice cream samples were tempered to -12±2°C and conducted in an isolated booth. For sensory assessment, samples were served in 50 ml cups (polystyrene) with three-digit codes. Evaluation of samples was carried out by a trained panelist (n=7) for appearance/color, body & texture, mouthfeel, and flavor on a 9-point hedonic scale whereas the overall acceptability score was the mean of all attributes (Stone & Sidel, 2004).

2.13. Optical microscopy and image analysis

An optical microscope was used at 10X magnification to visualize the air cells and ice crystals (Magnus, MIPS, Olympus, and CH20i) with slight modifications in the methods as described by Donhowe, Hartel, and Bradley (1991) and Chang and Hartel (2002). A small amount of ice cream sample was mounted on a pre-cooled (2 - 4°C) microscopic slide (equilibrated in a freezer by putting slides in a beaker containing ethanol) and dispersed as a thin layered smear using 1-2 drops of kerosene oil with another slide. Two separate slides were used to study air cell and ice crystal size at temperatures of -6°C and -15°C, respectively. The diameter of each air cell or ice crystal was measured using the Image J software.

2.14. Statistical analysis

Data were analyzed using Analysis of Variance (ANOVA) and the Duncan test differences among means by Statistical Package for the Social Sciences, SPSS 20.0 (SPSS Inc. Chicago, IL). Spearman was used to evaluating the correlation among variables. Multivariate exploratory techniques (PCA and cluster analysis) have been applied to investigate parameter correlation for analyzing the stabilizing mechanism of the formulation using Origin software, version 2019b.

3. Results and discussion

3.1. Physico-chemical composition of ingredients and ice cream

The milk sample had 4.5±0.1 % fat, 8.5±0.44 % SNF (solid not fat) and 0.17±0.02% titratable acidity. The butter contained 86±2.00% fat and 0.16±0.02% titratable acidity.

For the different treatments, total solids in the ice cream ranged from 36.71 to 36.77%, fat content from 11.01 to 11.09%, protein content from 5.31 to 5.37%, ash from 0.731 to 0.735%, pH from 6.50 to 6.70 and titratable acidity from 0.170 to 0.175% lactic acid. Chemical composition for all the treatments showed a statistically non-significant compositional difference ($p \geq 0.05$). The findings were consistent with Pagthinathan (2020), Pinto et al (2006), and Murtaza et al (2004) in plain ice cream of ginger processed products, ginger and fig paste ice cream, respectively. Our previous studies on Indian gooseberry and ginger ice cream also revealed similar results (Goraya & Bajwa, 2015; Goraya & Bajwa, 2018; Gabbi, Bajwa & Goraya (2018).

3.2. Cream stability

Cream stability of the mix reflects the performance of mix during quiescently frozen storage (Cheng et al., 2015). SM1 sample showed a minimum (4.48%) serum volume fraction percentage for the first two days which then increased drastically during the three-week storage. This observation could be attributed to weak protein hydrocolloid bonds. Cheng et al. (2015) also reported that weak bond formation can result in serum phase separation.

Cream stability of the treatments over a period of three weeks was compared using hierarchical cluster analysis (HCA) (Fig. 2). The distance linkage showed that the overall SM2 sample showed the least serum phase separation whereas the separation was highest in the SW2 sample. The control sample was consistently found in cluster 1 while other treatments moved between the clusters over a period of three weeks. As the storage period exceeds from the 2nd week, this trend changed and it was observed that cluster 1 got split into two subgroups (control and SW2, SM1, and SW1) whereas cluster 2 had SM2 sample which was found separately without any other subgroup correlation effect. The trends obtained in the third week clearly depicted that amalgamation order impacts the physical interactions within the ingredients and can improve the stability of the system which is desirable. Therefore, pre-treatment i.e. gelling with milk, and treatment i.e., adding butter prior to dry ingredients in SM2 sample played a synergistic role resulting in a most stable emulsion.

Syed et al. (2018) reported that homogenization impacts texture stability by destabilizing fat molecules in the mix. It was concluded that an ingredient will exhibit its functionality; however, it is their physical form, inclusion rate, time as well as processing that can significantly impact final product attributes and stability.

3.4. Viscosity

Changes in the amalgamation order of ingredients significantly ($p<0.05$) affected the mix viscosity. The maximum and minimum viscosity in SM1, and control were 8406.20 cP and 1490.02 cP respectively. Overall, SW samples had 0.7 to 0.8 times lower viscosity than SM samples and within treatments, treatment 1 with dry ingredient inclusion first had 2.6 to 2.9 times higher viscosity than treatment 2 samples with butter addition first.

Evidently, physicochemical properties depends on ingredients, their ratio and interactions during processing steps. The difference in the viscosity values of SM1 (8406.20 cP) and SW1 (6363.44 cP) indicate that milk components interact with sodium alginate forming stronger networks than water. Therefore, irrespective of the pre-treatment sodium alginate underwent, inclusion prior to butter addition resulted in higher viscosity values. These results support the hypothesis that alginate sodium ions ($Na+$) interacted with a calcium ion and formed cross-linked polymers having maximum stability leading to the higher viscosity of the ice cream mix (Waldman, Schechinger, Govindarajoo, Nowick, & Pignolet, 1998; Syed et al., 2018). Moreover, calcium ions greatly increased the viscosity that results in gelation by assimilating into a three-dimensional zigzag (egg-box model) structure (Imeson, 2010). Thus, due to intervention in alginate gel with milk and its interaction with ingredients amalgamation order, a strong irreversible gel formed which amplified viscosity of samples as shown in Table 1. The lower viscosity of SM2 and SW2 (SM2>SW2) samples as compared to SM1 and SW1 could be because of milk proteins that exhibited emulsification properties on fat when added to the system. It is evident that sodium alginate formed stronger cross-links with calcium in milk when compared with water.

It was established that SM1 and SW1 mixes were more viscous which might be due to the gelation action of milk protein. Gelation step, irrespective of the medium, enhanced the mix viscosity as compared to control which improved the texture and physical attributes of the ice cream (Syed et al, 2018).

The specific gravity was least (1.03) for control whereas it was the highest for SM1 (1.17) sample. Viscosity and specific gravity of mix exhibited a direct correlation (r^2=0.7972). This relationship has previously been reported by Pagthinathan (2020) and Gabbi et al. (2018) and Goraya and Bajwa (2015, 2018) in the ginger and amla ice cream.

3.5. Overrun

A significant ($p<0.05$) variation was observed in overrun values for all considered treatments. Before hardening, the lowest overrun was observed in the control sample (79.63%) and highest in SM2 (110.61%). Based on the differences in overrun, after hardening for overnight, a dramatic change in the overrun values of all samples was observed except for SM2 as shown in Table 1. Mix viscosity has an important role to play in determining the overrun capacity because if the viscosity of the mix is already too high such as in the SM1 sample, the whipping ability and overrun of respective mix decreases ultimately (Syed et al, 2018).

Thus, the mixes showed a noticeable difference in overrun depending on the gel-forming potential of sodium alginate (pre-treatment) and the method of mixing ingredients (treatment). Marshall, Goff, and Hartel (2003) also stated that hydrocolloids and fat structure have a major influence on air stability, thermodynamically. The control mix has a low potential for air holding capacity, but a transcendental in the ice crystal (Fig. 5) which can be seen in the microscopic assessment. This could be due to an inappropriate dispersion/stabilization of air cells, despite incorporation during whipping (Soukoulis et al. 2008). The intervention in the unification order of ingredients (treatment) and the formation of strong alginate gel with milk (SM) thereby affect the air holding capacity throughout the sample matrix. Owing to the properties of dairy protein emulsification, their interaction with other components at the air cell interfaces leading to different overrun values (Schmidt, 2004).

The relationship between overrun and viscosity is quadratic, i.e too low/ high viscosity resulted in an adverse effect on overrun whereas an appropriate viscosity can hold enough amount of air. If the mix is too thin, film around the air bubble drains rapidly (Clarke, 2004), if too viscous, it becomes difficult to agitate energetically and may preclude air incorporation with improper air distribution (Bahramparvar, Razavi, Tehrani, & Alipour, 2013). Thus, transitional viscosity resulted in proper agitation to hold an assured amount of air within the matrix of SM2 (Fig. 5). Adapa, Dingeldein, Schmidt & Herald (2000) also stated that a highly viscous system does not favor foaming capacity but does favor stability. Marshall & Arbuckle (1996) also stated that ice cream mixes with high viscosity showed limited whipping capacity.

The difference in the specific gravity of ice cream is significant ($p<0.01$). Control samples had a maximum specific gravity of 0.62 whereas minimum in SM2 sample (0.58) due to higher overrun value (Table 1). The similar result has been reported by Samahy, Youssef, and Moussa (2009), overrun value ice cream decreased from 55.71 to 43.11 % when specific gravity increased from 0.71 to 0.86 in cactus pear pulp. The correlation (r^2=-0.9859) between the overrun and specific gravity was negatively associated. Arbuckle (1977) also reported a negative relation of specific gravity with an overrun of ice cream.

3.6. Instrumental hardness

Pre-treatment and treatment significantly ($p<0.05$) influenced ice cream hardness. The results showed that the hardness of control was maximum (1353 g) and lowest in SM2 (818.67 g) among all samples. The manufacturing order of any process affects the chemical stability of the end product, as mixing order has a direct impact on the chemical reaction for the formation structure especially in ice cream (Schmidt, 2004). Ice aggregated in a continuous network type of ice phase alters the structure and makes it harder (Goff & Hartel, 2013) because ice crystal shape, size, and amount contribute towards hardness that is directly related to mix rheology (Muse & Hartel, 2004). Serum phase micro-viscosity and stability (SM) influences the non-crystallized form of water and prohibits ice crystal collisions from preventing ice recrystallization (Hagiwara & Hartel, 1995; Bolliger, Kornbrust, Goff, & Erich, 2000).

A negative (r^2=-0.8335) correlation was observed between overrun and hardness. The air cells contributed towards the lightness along with the storage integrity of the product. Sofjan and Hartel (2004) quantified that the light texture of ice cream was due to the air holding capacity of samples which have a direct impact on the hardness of ice cream. Enlarged ice crystal size in ice cream lead to the icy structure with coarseness (Sakurai, Hakamata, Tomita, & Yoshida, 1996; Muse & Hartel, 2004).

3. 7. Melting characteristics

The pre-treatment and treatment steps significantly ($p<0.05$) influenced the first drip time and melting rate of the ice cream samples (Fig. 3). The first drip time of SM2 samples was the longest (25.19 min) than all the samples and control took the least time (11.30 min) for its first drip (Fig. 3 (B)). The melting rate of all the samples varied significantly; the control melted about 89.67% in 100 min, followed by SW2 (64.98%), SM1 (59.31%), SW1 (53.65%), and the least in SM2 (49.23%) sample (Fig. 3(A)). The probable reason for the slow meltdown of the SM2 sample could be the variance in the air cell heat transfer rate, which acts as an insulator and stabilizes the sample. According to Soukoulis et al. (2008) and Sojjan and Hartel (2004), overrun is correlated with low heat transfer, as air cells act as insulators, resulting in low melting rates.

Although viscosity values followed an increasing trend from control SW2 < SM2 < SW1 < SM1, first drip loss followed an increasing trend from control < SW1 < SW2 < SM1 < SM2, and for melting rate it was SM2 < SW1 < SM1 < SW2 < control. But, it is not only the viscosity of the mix which controls the first drip loss and melting rate of the ice cream, but also fat destabilization and overrun which play an important role in determining the melting behavior of the ice cream (Marshall & Arbuckle, 1996). With regards to the relationship between fat destabilization (r^2=-0.963) and overrun (r^2=-0.655), they are negatively correlated with the melting rate. Similar trends have been reported by Muse and Hartel (2004), Goraya and Bajwa (2015; 2018) and Gabbi et al. (2018) for the viscosity relationship to the melting rate.

The authors also studied the correlation between melting rate, fat destabilization, and ice crystals. The fat destabilization rate and ice crystal size exhibited a linear negative and positive association with melting rate, respectively. The collective effect of fat destabilization (FD) and ice crystal (IC) on the melting rate is best described by the linear regression equation [5]:

$$\text{Melting rate} = 215.035\ (\text{FD})^{-0.995}(\text{IC})^{0.0453} \qquad (5)$$

The final defined correlation predicts the melting rate with a reasonable precision with an average deviation of 4.69% and the regression coefficient (r^2) of 0.96. The predicted value of 99 % is within ± 7% of the experimental value as explained in the equation.

3.8. Fat destabilization

Fig. 5 illustrates the internal structure of frozen treatment samples of ice cream from which it may be interpreted that SM pre-treatment i.e. alginate mixed with milk and SM2 pre-treatment i.e. butter addition prior to dry ingredients significantly and positively affected the gelling-agent induced aggregate formation within the product matrix and is the most suitable treatment in this study. Fat destabilization value for SM2 sample (19.69%) was 3.3 times higher than control (5.94%) with the least fat destabilization as shown in Fig. 3(C). It has been reported that the presence of large fat clumps, either present originally or formed initially, can rupture the air cells in ice cream. Therefore, fat destabilization is a desirable phenomenon for maintaining structural stability (Marshall et al., 2003).

The authors also studied the stability coefficient for all the samples (Fig. 3C). It was observed that fat destabilization and stability coefficient have a significant ($p<0.05$) inverse correlation (r^2=-0.99). The maximum stability coefficient was observed in control (94.06%) and the minimum was observed in the SM2 sample (80.27%). The samples with a higher stability coefficient delay the foaming ability of the mix by impeding the stability of the matrix. The results were concordant to Goff & Hartel (2013).

3.9. Instrumental color analysis

Table 2 outlines the L*, a* and b* values obtained for the different ice cream samples. The results revealed that the order of ingredient addition does not only impact the structural stability but also the physical attributes such as color of the ice cream samples.

L* value suggests the whiteness of the sample which varied significantly ($p<0.05$). L* of the control sample presented similar results (69.33) as reported earlier by Goraya and Bajwa (2015) indicating that L* value remains within this range, if a standard manufacturing protocol is used for ice cream manufacturing. The maximum L* value was observed in SM2 (70.15) which could be due to increased overrun values resulting in more air cells. Further, this hypothesis is confirmed by a positive correlation between overrun ($r^2=0.80$) and air cell size ($r^2=0.056$) with the L* value of the sample. Teh, Dougherty, and Camrie (2005) also reported that a higher overrun contributes to lightness in the sample.

Similar to the observations for L* values, interventiosn in alginate gel hydration and the amalgamation order of ingredients within the six ice cream samples led to significant differences ($p<0.05$) in a* and b* values. The a* value of the samples indicated a lower valye from control (-1.44) and ranged from -1.50 to -1.61 for the five treatments. Negative value of a* reflects greenness which might have been due to the newer ingredient interactions and modified structural network which highlighted different color attributes as compared to the control sample. It was also observed that b* value was positive indicating yellowness in al the samples, and its value heightened in the treatment samples within which SM2 and SW1 had highest and statistically similar scores. It is concluded that ingredient amalgamation order can significantly the color attribtues and its perception in the samples.

3.10. Sensory attributes

Among the sensory attributes, only appearance/color scores were somewhat similar, however, the rest of the attributes differed significantly ($p<0.05$) (Fig. 4) for the four formulations. We also found that appearance scores had a positive correlation value with L value measured using Mini Scan Xe Plus (0.95).

Pre-treatment, as well as treatment, significantly affected the body & texture, and mouthfeel of ice cream samples. The control sample received a minimum score (6.00) followed by SW2 whereas SM2 received a maximum score (8.00). The lower score for control and SW2 could be attributed to the high number of ice crystals which in turn were reflected as the increased hardness of samples and affected the body and texture of the sample by giving more cold sensation in the mouth. Literature studies revealed that the body and texture of the ice cream, directly influenced by ice shape and volume, govern overall acceptability recorded by panelists (Soukoulis et al., 2008; Lewis, 2007; Dogan & Kayacier, 2007). Lewis (2007) revealed that the mouthfeel is inversely proportional to the presence of large ice crystals in the ice cream and this was also observed in the present study wherein SM2 with smaller ice crystals was scored 8.00 for mouthfeel.

The flavor value of the SM2 (8.06) sample was significantly higher than all samples (Fig. 4) because the fat structure ameliorated the perception of ice crystals in the mouth. Varela, Pintor, and Fiszman (2014) also suggested that when small crystals (desired size for the frozen product) melted in the mouth, the smooth liquid is perceived as a creamy structure by panelists. A positive correlation (r^2=0.96) between overall acceptability and overrun was obtained. Adapa et al. (2000) also asserted that lower overrun capacity negatively affects the overall acceptability of samples.

3.11. Optical microscopy and image analysis

The maximum diameter of the air cell size was observed in SW1 (1 μm) and minimum in SM2 (0.90 μm). It was further confirmed that the order of addition of ingredients impacts the structural stability of the ice cream. Microscopic examination revealed that the larger air cells were characterized by the presence of weak boundaries and were observed in all the samples except SM2. The microscopic image (Fig. 5) showed a thick, fat coalesces around the droplets in the SM2 sample, followed by SM1.

The authors in this study observed that a viscosity value of approx. 3000 cP is desirable. The formulation, treatment process as well as viscosity are in coherence for the SM2 sample which prevented uniformly distributed small air cells from collapsing as they were enclosed in strong boundaries and surrounded by fat clusters attributed to a higher fat destabilization value. Goff & Hartel (2013) reported that air cells are prone to rupturing if large fat clumps were initially present or may be formed at the early stages of the freezing process. Overall, SM pretreated samples held air for long-duration as compared to

SW pretreated samples and authors believe that emulsifying properties of dairy proteins altered sodium alginate’s interaction with others leading to dispersibility and stability air cell network of ice cream (Schmidt, 2004).

With regards to ice crystals, Fig. 6 illustrated that the largest and smallest ice crystals had a maximum diameter in control samples whereas the SM2 matrix showed uniformly distributed smallest large and small ice crystals. Additives such as alginate used in ice cream formulation, interact with dairy proteins in oil-water and air-water interface, this interaction contributes to the fat emulsion destabilization leading to smaller ice crystals and desirable texture (Moon & Mangino, 2004). Among all the samples, it was observed that the control and SW2 exhibited dark, larger as well as a higher number of ice crystals. The SM1 sample demonstrated medium-sized, light-dark colored crystals while SW1 had smaller but darker ice crystals than the SM2 sample. It is further confirmed by the statistical analysis which revealed a negative correlation (r = -0.95) between ice crystal size and overall acceptability. Increased ice crystal size could have resulted in a harder ice cream structure, lower scores for the sensory attribute, and melting stability of ice cream (Sakurai et al., 1996; Muse & Hartel, 2004). Interestingly, ice crystals' size had an influence on air cell stability directly impacting the physical characteristics and structural elements of ice cream (Sofjan & Hartel, 2004).

3.12. Correlation of sensory attributes with both physical and instrumental properties, along with discrimination of the process intervention and ingredient unification.

The relationship between physical and instrumental properties was analyzed using a PCA biplot (Fig. 7). The biplot revealed that the projection of the main two main components (PCs) covered 79.66% and 12.55% of the total variance, respectively, of PC1 and PC2 and grouped to four quadrants of the matrix leads to the following suggestions:

1. As per the PCA plot, the hardness and melting rate (M_{100}) have a direct influence on the control and SW2 sample and seemed to have more ice crystals. The melting rate of these samples was higher as small and large ice crystals contributed to decreased serum concentration (Soukoulis et al., 2008)

2. The maximum air cell diameter was observed to be correlated positively with SM1 and SW1, but the overrun value was negatively correlated. Interestingly, the average diameter of large and small air cells in SM1 and SW1 was found to be higher than the SM2 sample. Therefore, according to the PCA biplot, the SM2 sample (milk-based gelation) had the highest overrun with pronounced air cell distribution, resulting in good quality body & textured ice cream.

3. Fat destabilization and overrun were found to be positively associated with sensory attributes for SM2 in the PCA biplot statistical. Because of a high-fat destabilizing value, ice crystal structure as well as high serum phase concentration resulted in a low melting rate (SM2) and highly acceptable mouthfeel along with flavor scores, among all samples. Therefore, an ice cream sample (SM2) in which sodium alginate was hydrated in milk and butter was added prior and achieved maximum sensory attribute scores with the least developed coarse and icy defect.

4. Conclusion

The stabilizing system with alginate gel hydrated in milk, followed by butter-dry ingredients addition (SM2) was the best sample among the different samples. The gelation interactions in the SM2 sample, among others, led to a reinforcement of rheological and structural parameters, as well as to the improvement in the stabilizing effect (such as air holding capacity, strong emulsion boundaries, and small ice crystals) under quiescent frozen storage conditions. SM2 sample exhibited high overrub and microstructural studies revealed lamellar distribution around the air bubbles preveting their rupturing. The PCA biplot score correlated a phenomenal impact on ice cream structural elements of SM2 as better, among all other samples based on sensory, fat destabilization, air cell, overrun, and melting resistance. The outcomes of this investigation concluded that sodium alginate gel formed with milk followed by butter-dry ingredients addition forms a stabilized ice cream structure for long-term storage.

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Figure Legends

Fig.1. Process flow chart for development of ice cream mix. Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Fig. 2. Cream stability of ice cream samples, Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion, using hierarchal cluster analysis (Wards' Method).

Fig. 3. Melting rate (A), first dripping time (B), fat destabilization & stability coefficient (C) of ice cream samples, Control (■, C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (▼, SW1) and milk (●, SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (♦, SW2) and milk (▲, SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Fig. 4. Sensory attributes of ice cream samples, Control (▬ ● ▬, C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (▪▪▪●▪▪▪●, SW1) and milk (━━ ▪ ━━, SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (▬●▬, SW2) and milk (═══, SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Fig. 5. Air cell size (XXX, large and ═ small) and microscopic structure of ice cream samples, Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Fig. 6. Ice crystal size (XXX, large and ═ small) and microscopic structure of ice cream samples, Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Fig. 7. Principal component analysis (PCA) biplot of sensory attributes with physical and instrumental properties. Overrun (OR), instrumental hardness (HD), melting rate (MR_{100}), fat destabilization (FD), lightness (L), air cell size (LAC and SAC) and ice crystal (LIC and SIC), and sensory attributes (appearance/color (App), body & texture (BT), mouthfeel (M), flavor (F) and overall acceptability (OA)

Table legends:

Table 1: Formulation of ingredients based on pre-treatment and treatment of the ice cream samples.

Table 2: Physical/structural properties of ice cream prepared by amending alginate gel formation and ingredients amalgamating order.

Table 1 Formulation of ingredients based on pre-treatment and treatment of the ice cream samples

Sample Name	**Pre-treatment**			**Treatment**				
	Sodium alginate, g	**Milk, ml**	**Water, ml**	**Milk, ml**	**White Butter, g**	**SMP, g**	**Sugar, g**	**GMS, g**
Control	17.5	x	x	3507.5	460	257.5	750	7.5
SW_1	17.5	x	300	3507.5	460	257.5	750	7.5
SW_2	17.5	x	300	3507.5	460	257.5	750	7.5
SM_1	17.5	300	x	3507.5	460	257.5	750	7.5
SM_2	17.5	300	x	3507.5	460	257.5	750	7.5

Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

Table 2 Amendment in alginate gel formation and ingredients amalgamating order effect on physical/structural properties of ice cream

Samples	**Viscosity**[#] **(cP)**	**Specific gravity**[#]	**Overrun (%)**		**Specific gravity**	**Hardness /texture (g)**	**Color attributes**		
			Before hardening	**After hardening**			**L***	**a***	**b***
C	1490.02^{e} ±5.71	1.03^{c} ±0.02	79.63^{c} ±0.31	46.52^{d} ±0.07	0.62^{a} ±0.003	1353.00^{a} ±56.24	69.33^{c} ±0.17	-1.44^{a}± 0.04	3.02^{c}± 0.07
SM_1	8406.20^{a} ±4.05	1.17^{a} ±0.07	90.63^{b} ±0.60	53.59^{c} ±0.08	0.58^{b} ±0.004	923.33^{c} ±28.43	69.74^{b} ±0.10	-1.56^{c}± 0.02	3.13^{b}± 0.06
SM_2	3181.83^{c} ±5.84	1.11^{ba} 0.02	110.61^{a}± 0.68	102.08^{a} ±0.16	0.49^{c} ±0.006	818.67^{e} ±23.46	70.15^{a} ±0.06	-1.61^{c}± 0.04	3.58^{a}± 0.03
SW_1	6363.44^{b} ±3.77	1.12^{ba} 0.03	93.66^{b} ±0.41	60.71^{bc} ±0.13	0.55^{b} ±0.003	916.33^{d} ±25.15	70.03^{a} ±0.06	-1.60^{c}± 0.02	3.55^{a}± 0.02
SW_2	2207.4^{d} ±2.11	1.07^{bc}±0.02	85.09^{bc} ±0.69	50.16^{cd} ±0.11	0.61^{a} ±0.005	1160.00^{b} ±25.71	69.52^{bc} ±0.19	-1.50^{b}± 0.02	3.07^{bc}± 0.06

Data were expressed as means ± SD of triplicate readings, means with different (a,b,c,d,e)superscript in the same column differs significantly ($p<0.05$), #mix

Control (C) refers to the standard dairy-based ice cream mix formulation; sodium alginate hydrated in water (SW1) and milk (SM1) mixed in the milk to which dry ingredients were added before butter addition; sodium alginate hydrated in water (SW2) and milk (SM2) mixed in the milk to which butter was added before dry ingredients inclusion.

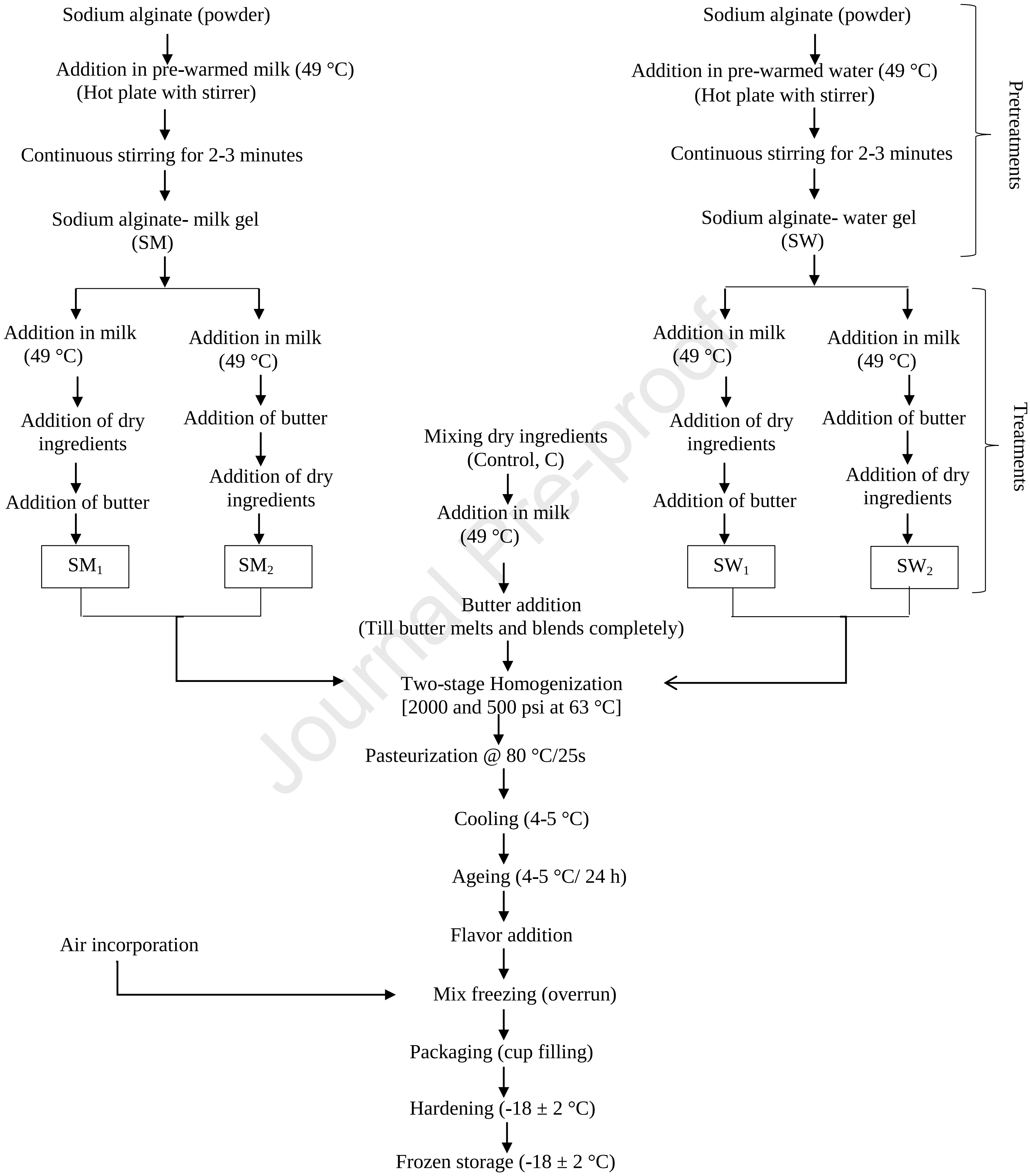
Sodium alginate (powder)
Addition in pre-warmed milk (49 °C)
(Hot plate with stirrer)
Continuous stirring for 2-3 minutes
Sodium alginate- milk gel
(SM)
Sodium alginate (powder)
Addition in pre-warmed water (49 °C)
(Hot plate with stirrer)
Continuous stirring for 2-3 minutes
Sodium alginate- water gel
(SW)
Pretreatments
Addition in milk
(49 °C)
Addition of dry
ingredients
Addition of butter
SM_1
Addition in milk
(49 °C)
Addition of butter
Addition of dry
ingredients
SM_2
Mixing dry ingredients
(Control, C)
Addition in milk
(49 °C)
Addition in milk
(49 °C)
Addition of dry
ingredients
Addition of butter
SW_1
Addition in milk
(49 °C)
Addition of butter
Addition of dry
ingredients
SW_2
Treatments
Butter addition
(Till butter melts and blends completely)
Two-stage Homogenization
[2000 and 500 psi at 63 °C]
Pasteurization @ 80 °C/25s
Cooling (4-5 °C)
Ageing (4-5 °C/ 24 h)
Flavor addition
Air incorporation
Mix freezing (overrun)
Packaging (cup filling)
Hardening (-18 ± 2 °C)
Frozen storage (-18 ± 2 °C)

Fig. 1.

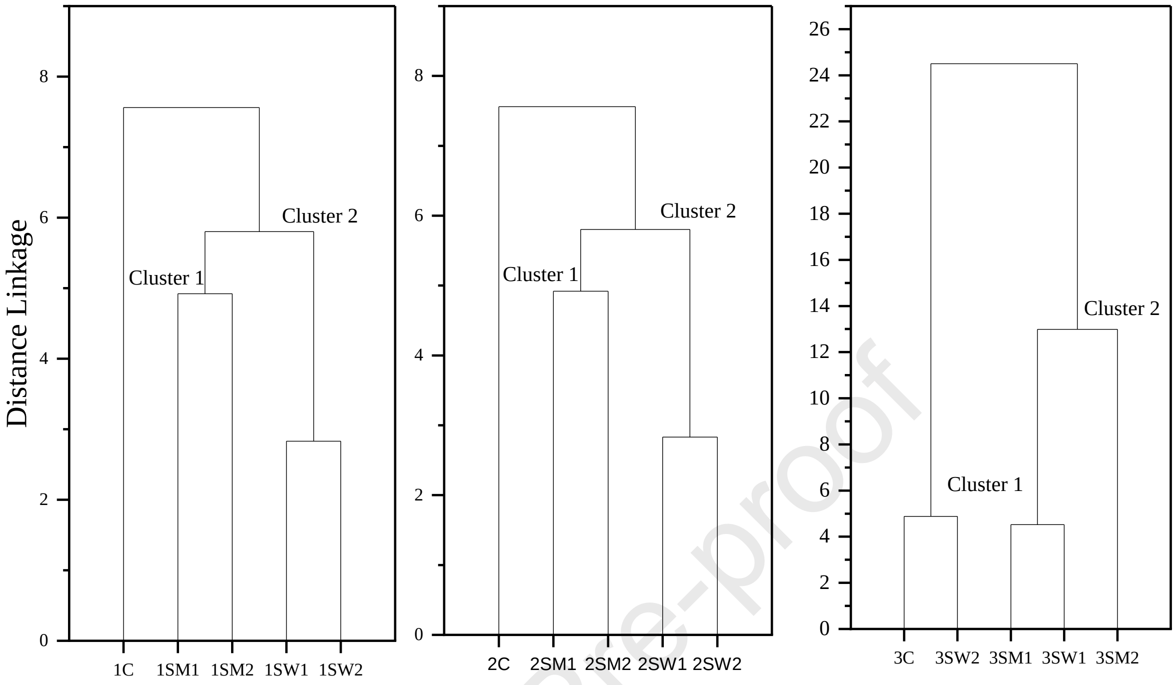
Distance Linkage
0
2
4
6
8
Cluster 1
Cluster 2
1C
1SM1
1SM2
1SW1
1SW2
0
2
4
6
8
Cluster 1
Cluster 2
2C
2SM1
2SM2
2SW1
2SW2
0
2
4
6
8
10
12
14
16
18
20
22
24
26
Cluster 1
Cluster 2
3C
3SW2
3SM1
3SW1
3SM2

Fig. 2.

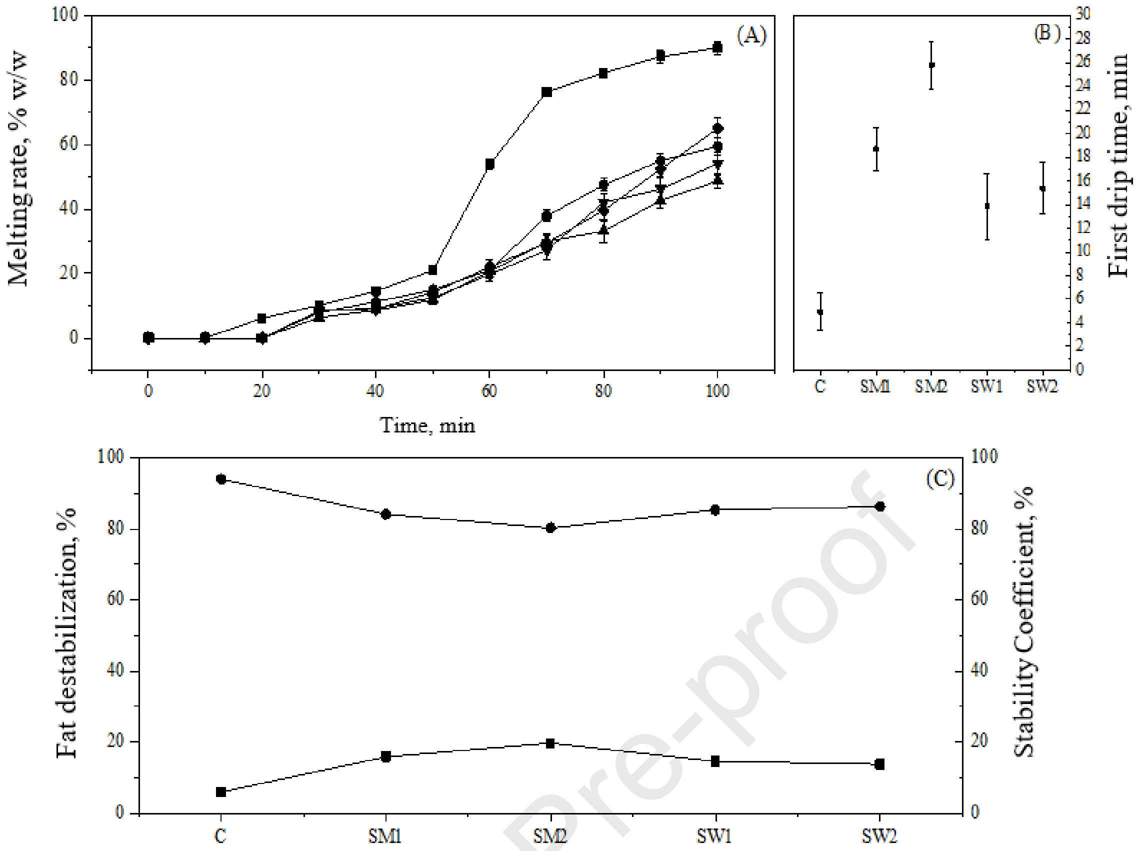
(A)
Melting rate, % w/w
Time, min
0
20
40
60
80
100
(B)
First drip time, min
C
SM1
SM2
SW1
SW2
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
(C)
Fat destabilization, %
Stability Coefficient, %
C
SM1
SM2
SW1
SW2
0
20
40
60
80
100

Fig. 3.

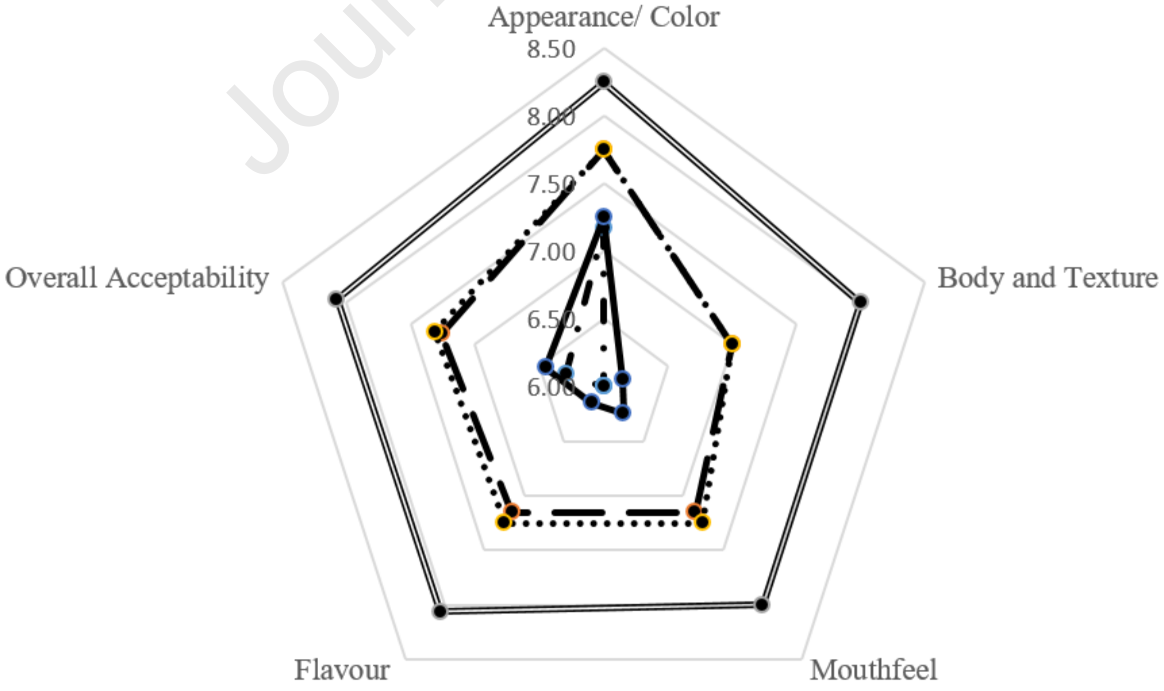
Appearance/ Color
Body and Texture
Mouthfeel
Flavour
Overall Acceptability
8.50
8.00
7.50
7.00
6.50
6.00

Fig. 4.

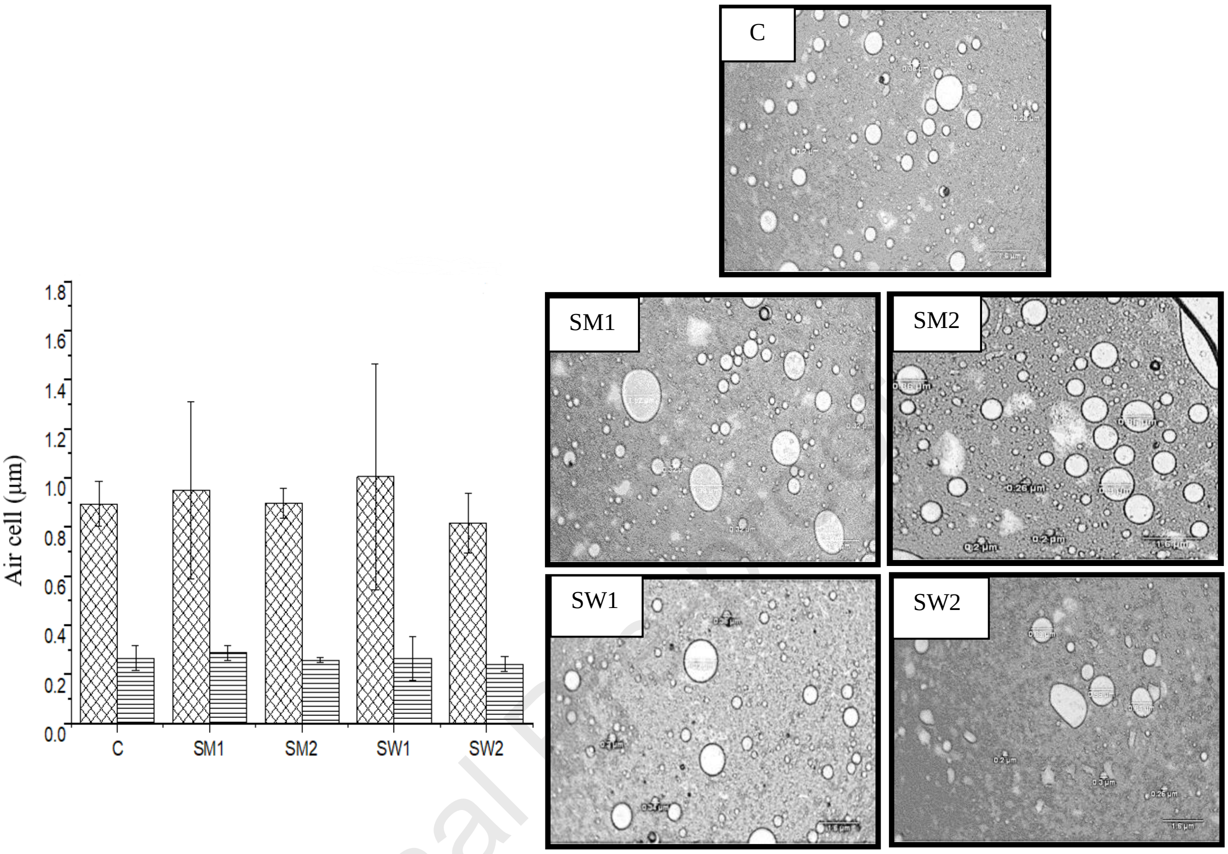
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
Air cell (µm)
C
SM1
SM2
SW1
SW2
C
SM1
SM2
SW1
SW2

Fig. 5.

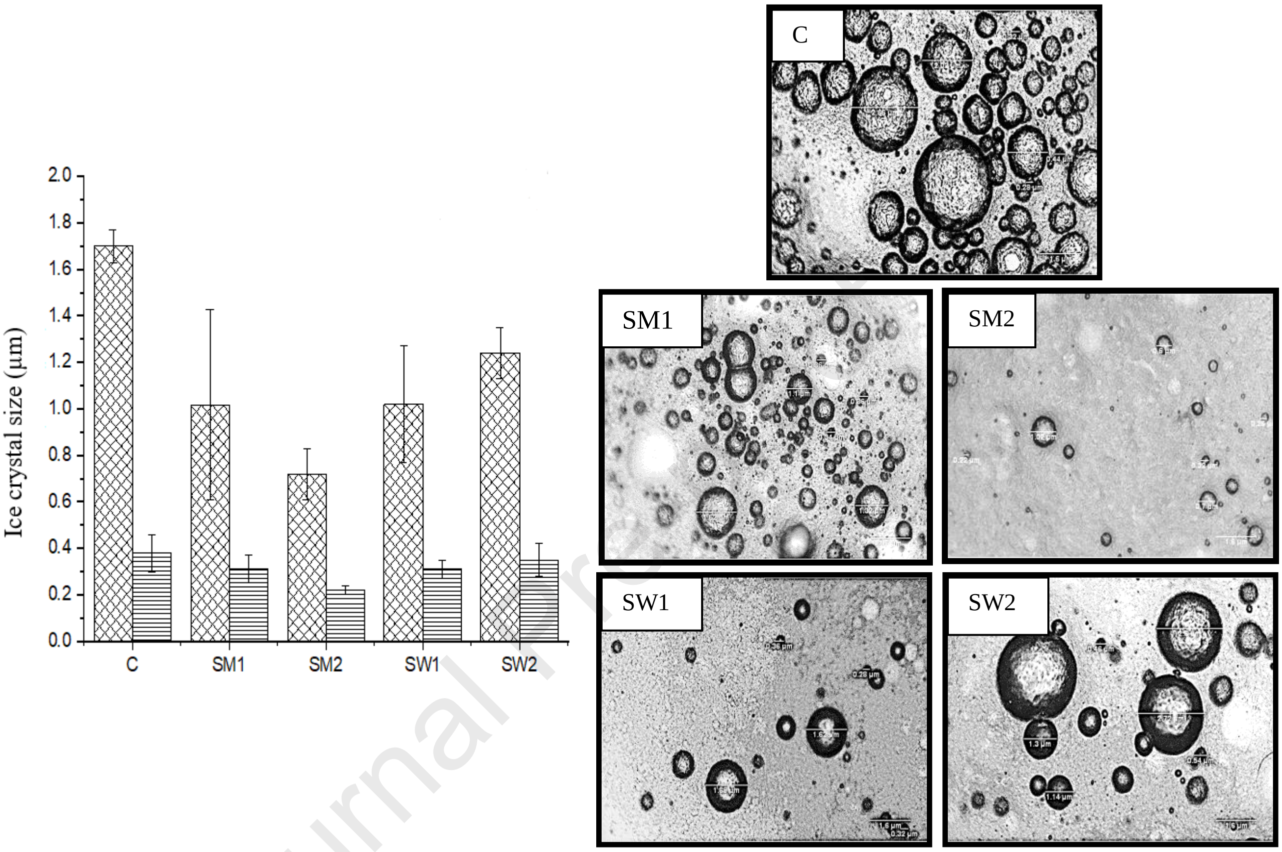
Ice crystal size (μm)
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
C
SM1
SM2
SW1
SW2
C
SM1
SM2
SW1
SW2

Fig. 6.

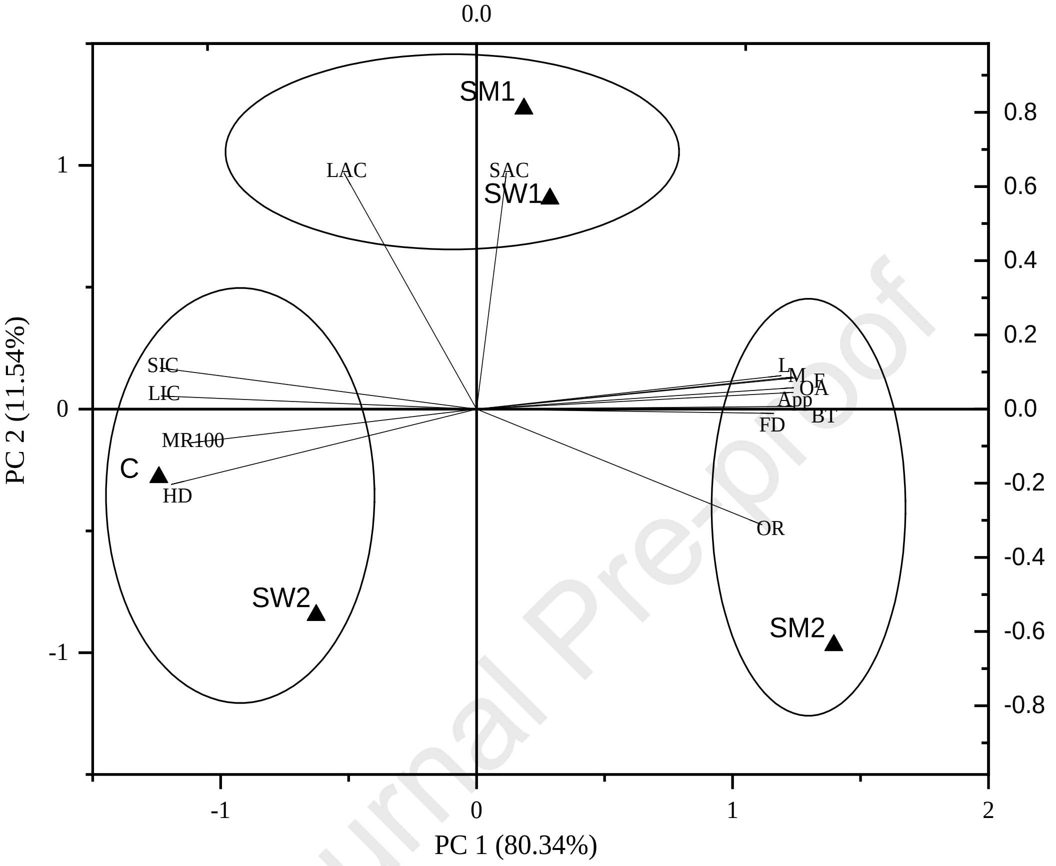
0.0
SM1
LAC
SAC
SW1
SIC
LIC
MR100
C
HD
SW2
L
M
F
OA
App
BT
FD
OR
SM2
1
0
-1
0.8
0.6
0.4
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1
0
1
2
PC 1 (80.34%)
PC 2 (11.54%)

Fig. 7.

Highlights:

- A standard ice cream recipe was reformulated by amalgamating ingredient addition order.
- Water and milk based sodium alginate gels were developed and used for mix development.
- Physico-chemical properties, sensory, microscopic, and melting characteristics of ice cream samples was studied.
- Reformulation can yield a mix with high stability and overrun, smaller ice crystals and more destabilized fat.

From:
Shivani Pathania
Teagasc Food Research Centre, Ashtown
Dublin-15, Ireland

To,
Editor-in-chief,
LWT- Food Science & Technology,

Subject: Regarding conflict of Interest for the research article ‘Impact of sodium alginate gelling and ingredient amalgamating order on ingredient interactions and structural stability of ice cream’

Sir,

Please allow me to declare that there is no conflict of interest for this article.

Yours Sincerely,

Shivani

Shivani Pathania

Dated: 3rd February, 2021